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# Enhanced vacuum glazing bonding strength by anodic bonding assisted sealing method

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**Abstract:** A novel sealing method of vacuum glazing is introduced in this study. Anodic bonding technology was used to enhance the bonding strength of the sealant of vacuum glazing. Two sheets of plane glass were sealed by  $V_2O_5$ - $P_2O_5$ - $TeO_2$  glass powder with low glass transition temperature ( $339.3^\circ\text{C}$ ) by using anodic bonding assisted low-temperature sintering. Through analyzing the results of Shear Strength test, the diagram of micro-structures of the sealing bond taken by a micro Super-depth Microscopy, Scanning Electron Microscope (SEM), and Energy Dispersive Spectrometer (EDS), the best bonding treatment schedule was determined for achieving the strongest bond. The experiment results show that anodic bonding has a dramatic influence on bonding performance. When the sealing temperature and bonding time are kept constant, the shear strength of the sample increases with increasing the bonding voltage. With the bonding temperature of  $470^\circ\text{C}$  and the voltage of 600V, the air tightness of the sealed sample is  $3.9 \times 10^{-9} \text{ Pa} \cdot \text{m}^3/\text{s}$ . The

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improved bonding strength can enhance life-time of vacuum glazing.

**Key word:** Vacuum glazing; Anodic bonding; Low-melting glass; Bonding strength;  $V_2O_5$ - $P_2O_5$ - $TeO_2$ .

## Introduction

Recent reports released by International Energy Agency (IEA) show that buildings consume approximately 40% of total world energy in 2014<sup>[1]</sup>. Among all building fabric components, window and other glazed area are the weakest in terms of thermal insulation and are often referred as heat sink<sup>[2]</sup>. Therefore, reducing energy consumption through windows is the key to cutting building energy consumption.

Vacuum glazing that has excellent thermal and sound insulation properties, can potentially reduce heat loss through building windows and walls. Vacuum glazing consists of two sheets of plane glass, separated by a narrow vacuum gap with an array of support pillars, contiguously sealed together around their peripheries<sup>[3-5]</sup>. The schematic diagram of vacuum glazing is shown in Fig 1. The role of the vacuum gap between the two glass sheets is to eliminate the conduction and convection across the glazing<sup>[6,7]</sup>. In addition, when the Low-E coatings are applied on the internal glass surfaces within the vacuum gap, the thermal transmittance  $U$ -value of the vacuum glazing can be reduced to less than  $1.0 W \cdot m^{-2} \cdot K^{-1}$  <sup>[8-10]</sup>. Vacuum glazing was first patented by Zoller in 1913<sup>[11]</sup>. After about 80 years later, Collin and Robinson firstly reported successful vacuum glazing sample from the University of Sydney in 1991<sup>[12]</sup>. Since the vacuum glazing appeared in the market, it has attracted more and more attention due to its excellent thermal and sound insulation performance. However, the way for improving the life-cycle of vacuum glazing and reducing its fabrication cost is still a great challenge. Since the bonding strength of vacuum glazing plays an important role in the life-cycle of vacuum glazing, improving the bonding strength is a crucial matter for improving the durability performance of vacuum glazing. Novel bonding method to form the sealant of vacuum gap can improve the fabrication method of vacuum glazing, which is critical to address the issues of life-cycle and fabrications of vacuum

glazing.

The fabrication methods of vacuum glazing can be divided into two ways: pump-out method and vacuum chamber method<sup>[4]</sup>. The pump out method is a two-stage method. At the first stage, the edge seal is formed in a vacuum chamber. At the second stage, a high vacuum between the glass panes is created using a turbo-molecular pump within a conventional oven. Vacuum chamber method<sup>[23]</sup> is a one-stage method. Both “edge seal formation” and subsequent “pumping-out” processes are completed simultaneously within a vacuum chamber. There is not a pump-out tube in the vacuum glazing. Compared to pump-out method, vacuum chamber method is simpler and quicker. Since there is not a pump-out tube in the vacuum glazing, the appearance of this vacuum glazing is esthetically nicer.

Anodic bonding technique was firstly introduced by Wallis and Pomerantz in 1969, which can produce strong mechanical connection<sup>[13]</sup>. Anodic bonding technique has several advantages, for instance low bonding temperature, quick response and good sealing performance<sup>[14,15]</sup>. Traditionally, anodic bonding is widely used in production of numerous Micro Electro-mechanical systems (MEMS), such as electronics and semiconductor packaging industry<sup>[16,17]</sup>. Anodic bonding method belongs to the vacuum chamber method. Compared to the traditional vacuum chamber method, anodic bonding method has a dramatic influence on bonding performance. Lu J<sup>[18]</sup> utilized anodic bonding technique which employs a tin solder alloy containing an activating metal to seal the vacuum gap. It dramatically increases the bonding strength. However, to date no work about anodic bonding technique employing solder glass to seal vacuum glazing was reported.

Vacuum glazing sealed in the bonding furnace should maintain shrinkage during the cooling progress. The shrinkage progress could generate stress on the sealing edge of vacuum glazing which could make the sealing interface break. Therefore, the thermal expansion coefficient of solder glass must match the plane sheet. In this study,  $V_2O_5$ - $P_2O_5$ - $TeO_2$  low-melting glass is chosen as the edge seal material based on the early

research of our group<sup>[19]</sup>. This glass has expansion coefficient of  $83.5 \times 10^{-7}/^{\circ}\text{C}$ , which is close to that of glass sheet; low glass transition temperature of  $339.3^{\circ}\text{C}$  with a short phase changing period; and sealing temperature between  $430^{\circ}\text{C} \sim 470^{\circ}\text{C}$ . which is much lower than that of glass sheet. These properties makes the  $\text{V}_2\text{O}_5\text{-P}_2\text{O}_5\text{-TeO}_2$  low-melting glass perfectly to be the sealant of vacuum gap of vacuum glazing. In this work, it is the first time that anodic bonding technology was used to seal the vacuum glazing. A firm bonding strength and fine surface quality between plane sheet and solder glass were achieved in this work, thus it provides important theoretical basis and guidance for manufacturing and industrialization of the vacuum glazing and its application..

## Experiment

$\text{V}_2\text{O}_5\text{-P}_2\text{O}_5\text{-TeO}_2$  low-melting glass was chosen as the edge seal material. The compositions and various performance indexes of  $\text{V}_2\text{O}_5\text{-P}_2\text{O}_5\text{-TeO}_2$  glass are present in Table 1. The  $\text{V}_2\text{O}_5\text{-P}_2\text{O}_5\text{-TeO}_2$  low-melting glass was prepared using the melt quenching technique. The stoichiometric amounts of  $\text{V}_2\text{O}_5$  ( $\text{NH}_4\text{VO}_3$ ),  $\text{P}_2\text{O}_5$  ( $\text{NH}_4\text{H}_2\text{PO}_3$ ),  $\text{B}_2\text{O}_3$  ( $\text{H}_3\text{BO}_3$ ),  $\text{TeO}_2$ ,  $\text{ZnO}$  and  $\text{Na}_2\text{O}$  ( $\text{Na}_2\text{CO}_3$ ) (AR grade) was thoroughly mixed in a mortar. Firstly the reaction mixture was calcined at  $210^{\circ}\text{C}$  for 1 hour, and then at  $1150^{\circ}\text{C}$  for 2 hours in a high-purity alumina crucible. Secondly, the homogeneous melt was cast in a preheated graphite and annealed at  $350^{\circ}\text{C}$ . Annealing was carried out for a period of one hour to remove the thermal stress. Finally, the glass bulk was kept in a desiccator.

The prepared glass bulk was grounded into powder and sifted out through 500-mesh sieve. The binder (ethyl cellulose) was dissolved thoroughly in the solvent of terpineol and diethylene glycol butyl ether at  $80^{\circ}\text{C}$ . The glass powder was stirred homogeneously in the prepared solvent to obtain the glass paste. After this process, the glass paste was coated with screen printing and the coating was dried at  $150^{\circ}\text{C}$  to volatilize the solvent. Fig 2 shows the sealing progress of vacuum glazing. Bonding experiments were performed in an

anodic bonding furnace (BFS-1. Weina, China). The bonding pair was heated and a DC voltage was applied to the electrodes, to ensure a positive electrode potential on cathode side with respect to the glass. All anodic bonding processes were carried out under  $1.0 \times 10^{-3}$  Pa. The bonding temperature was set to a value between 430-470°C. The bonding voltage of 0-600V was applied, and bonding time was 10-20min.

One glass sheet with the thickness of 4mm was cut to the size of 20mm x 20mm. Then it is coated with glass coating. Following this process, they were superimposed and sealed. In the last step, the shear strength of the sealed samples was tested by using an in-house shear testing machine. Ultra-depth three-dimensional microscope and QUANTA FEG 450 field emission environment scanning electron microscopy (SEM) were used to observe morphology of the sealing sample. Assisted by Energy Dispersive Spectrometer (EDS), the composition and content of the micro-zone of the material were analyzed. The leakage rate of the samples was tested by using VARUA VS BR15 helium mass spectrometer. The criterion of leakage rate is less than  $5.0 \times 10^{-8} \text{ pa} \cdot \text{m}^3/\text{s}$

## Result and discussion

The main process parameters of anodic bonding refer to bonding temperature, bonding time and bonding voltage. The aim of controlling bonding temperature, bonding time and voltage is to transfer conductive ions in glass layer to establish necessary electrostatic field. Table 2 is the shear strength of samples at different bonding temperature. With the bonding temperature of 430°C, the bonding time of 10min and the bonding voltage of 400V, the shear strength is at the lowest level, approximately 0.621MPa. For the same bonding time, with increasing the bonding temperature, the shear strength of the sample increases. With the bonding temperature increasing to 470°C, the shear strength increases to 1.748MPa, which is three times larger than that when the bonding temperature is 430°C.

Fig 3 compare the shear strength of the samples at different bonding temperatures. With the bonding time of

10min, the rate of growth of shear strength when the temperature increases from 430°C to 450°C is less than that when the bonding temperature increases from 450°C to 470°C. Fig 4 presents the pictures taken by an Ultra-depth three-dimension microscope at different bonding temperatures. Fig 4 shows that as the bonding temperature increases, the surface of the bonding layer becomes more compact. When the bonding temperature was 430°C, some glass powder has not yet melted because the temperature of connection layer has just reached the softening temperature of solder. During the melting period, some particles migrate to the interface and form a reactive wetting layer on the interface, but the migration of particles is slow due to the low sealing temperature and the impedance of the solid grain. At this moment, the wetting phenomenon is not obvious, and the homogeneity of fusion solder is poor. Therefore, the surface of low melting glass layer is not compact and the bonding strength is poor. With the bonding temperature of 450°C, the melting rate of glass powder increases. And the increase in the bonding temperature accelerates the element migration, enables an obvious wetting phenomenon and better homogeneity of fusion solder. When the bonding temperature increased to 470°C, most glass powder melted. With the temperature increasing, the wetting phenomenon and homogeneity of fusion solder is better. So, the low melting glass layer is more compact and bonding strength is better. In addition, anodic bonding is an electrochemical reaction. With the bonding temperature increasing, the electrochemical reaction and the diffusion of charged species was accelerated, which accelerates the formation of the intermediate composition. Therefore, the bonding strength increases with the bonding temperature increasing.

Bonding time is also an important parameter in anodic bonding. Table 3 shows the shear strength of the samples at different bonding times. With the bonding voltage of 600V, the bonding temperature of 450°C, the bonding time of 10min, the shear strength is 1.491MPa. However, with the bonding time of 20min, the shear strength improves to 2.685MPa, approximately twice as that with the bonding time of 10min. Fig 5 compares the shear strength of the samples at different bonding times. With the same bonding temperature, the shear strength of the samples with the

bonding time of 20 minutes are higher than that with the bonding time of 10 minutes. Fig 6 presents the picture taken by using a three-dimension microscope at different bonding times. Fig 6 shows that when the bonding voltage and bonding temperature was the same, as the bonding time increases, the low melting glass layer become smoother and more compact. When the bonding temperature and bonding voltage is same, the migration rate of particles is constant. But as the bonding time prolongs, the total number of migrating particles increases. Therefore, the homogeneity of fusion solder will be better which leads to the low melting glass layer becoming smoother and more compact. Moreover, with the bonding time increasing, the electrochemical reaction and the diffusion of charged species will react more completely, enabling the intermediate composition layer to distribute continuously on the interface with certain thickness. As a result, the bonding strength increases with the bonding time increasing.

~~The main reason that the bonding strength is improved by extending the bonding time is that anodic bonding is an irreversible electrochemical reaction. Once the bond is formed, it is very difficult to break it. In addition, as the bonding time prolonging, the low melting glass can be fully sinter which leads to the low melting glass layer becoming smooth and compact. Therefore, prolonging the bonding time improves the bonding strength.~~

Table 4 shows the shear strength of the samples at different bonding voltage. With the bonding time of 20min, the sealing temperature of 430°C, the bonding voltage of 0V, the shear strength is 0.192MPa. However, when the bonding voltage is changed to 600V, the shear strength is increased to 1.793MPa, approximately nine times larger than that when no voltage was applied at the sample. Fig 7 compares the shear strength of the samples at different bonding voltages. With the same sealing temperature and bonding time, the shear strength of samples with no voltage applied, is lower than those with the voltage applied. Therefore, as bonding voltage increase, the shear strength of the samples increases and the bond is reinforced.

The microstructure and the element distribution at the bonding interface of the samples are investigated by SEM and EDS. Fig 8(a) presents the cross-sectional micrograph of the sealing sample taken by SEM with bonding



temperature of 450°C, bonding time of 20min, bonding voltage of 0V. Fig 8(a) shows that there are many cracks in the low-melting glass coating. The bond between low-melting glass coating and the plane sheet is weak. The gaps between the bonding layers can be seen. Fig 8(b) shows the cross sectional micrograph of the sample taken by SEM at 450°C, with bonding time of 20min and bonding voltage of 600V. Compared to Fig 8(a), the low-melting glass coating in Fig 8(b) is more compact, and no gap can be seen between the bonding layers. It is because when the sealing temperature is 450°C, the glass powder is not completely melted, then a little un-melted glass powder is unevenly distributed in the low-melting glass coating layer, which lead to the inconsistance of the expansion coefficient of low-melting glass coating. Consequently when the sample is cooled, many cracks in the low-melting glass coating and gaps between the bonding layers occur. With the bonding voltage of 600V, under the action of voltage, the ions in the glass migrate, which accelerates the flow of glass powder's particles and makes the expansion coefficient of low-melting glass coating more homogeneous. Otherwise (The last sentence is not very clear. Why do you use the word "Otherwise"), an intermediate composition was formed between low-melting glass coating and plane sheet, which makes the connection of these two layers stronger, so the gap between two layers disappeared.

Fig 8(c) shows the EDS analytical chart of the low-melting coating ( Fig 8(c) ① and Fig 8(c) ③) and plane glass sheet ( Fig 8(c) ② and Fig 8(c) ④). The main peaks in the Fig 8(c) ① stand for V and P, which are the main elements of the low-melting point glass. The main peaks in the Fig.8(c) ② stand for Si and Ca, which are the main elements of the plane glass sheet. With no voltage applied, the bonding between the low-melting glass coating and plane glass sheet exhibits wetting behavior. Compared to Fig 8(c) ①, the main peaks in the Fig 8(c) ③, apart from the peaks of V and P, also the Si peaks. In Fig 8(c) ④, the main peaks include V, P and Te peaks which are the unique elements of low-melting glass. This indicates that when the samples are subject with a voltage, the elements exchange happens across the interface between the low melting point glass coating and the plane glass sheet.

In order to assess whether a new bond has been formed at the interface between the low-melting point glass coating and plane glass sheet, the EDS linear distribution analysis of the major element along the straight line at the cross-sectional regions in SEM micrograph of Fig 9(a) is presented in Fig 9(b). The curve of each elements indicates that an intermediate composition layer has been formed at the interface between the low-melting glass coating and plane glass sheet. Therefore, when the samples are applied with a voltage, a new bond is formed between low-melting glass coating and plane sheet, which makes the bonding strength stronger.

The new bond formation entails two processes, an electrochemical reaction and the diffusion of charged species (anions and cations) at the interfaces between the solder glass and the plane glass sheet<sup>[20]</sup>. When the sample was applied with the voltage, the atoms of the solder glass at the interface between the low-melting point glass coating and plane glass sheet experienced an electrochemical potential. The metal oxide splits up in metal cations (such as  $V^{5+}$ ,  $Te^{4+}$  and  $Na^{+}$ ) and oxygen ions ( $O^{2-}$ ). The bond formation was attributed to the permeation of metal cations ( $V^{5+}$ ,  $Te^{4+}$  and  $Na^{+}$ ) into the plane sheet and the diffusion of oxide in the form of bridged  $O^{2-}$  toward the anode. Finally, an intermediate composition was formed at the interface between low-melting point glass coating and plane glass sheet. Fig 9(a) and Fig 9(b) show that there are trace of  $V^{5+}$  and  $Te^{4+}$  ion at the interface of the plane glass sheet, they are the unique element of low-melting point glass. It is speculated that there may be  $O^{2-}$  and  $V^{5+}$  or  $Te^{4+}$  forming a V-O or Te-O bond at the 1st-layer position, and Si-O bond is formed at the 2nd-layer of the interface between the coating and plane glass sheet. With increasing the sealing temperature, the permeation of metal cations and the diffusion of oxide in the form of bridge  $O^{2-}$  toward the anode are accelerated, which accelerates the formation of the intermediate composition. The strength of the new bond will be enhanced. The schematics of the process of anodic bonding is show in Fig 10.

In the experiments, with bonding temperatures of 470°C and 450°C, bonding time of 20min, and bonding voltages of 600V, the samples were tested for leakage rate. it the bonding temperature of 450°C, the bonding time

of 20min and the bonding voltages of 600V, the average leakage rate is  $7.0 \times 10^{-7} \text{ Pa} \cdot \text{m}^3/\text{s}$ , which is higher than the criterion ( $5.0 \times 10^{-8} \text{ Pa} \cdot \text{m}^3/\text{s}$ ).<sup>[21]</sup> With the bonding temperature of 470°C, when the other conditions being the same, the average leakage rate is  $3.9 \times 10^{-9} \text{ Pa} \cdot \text{m}^3/\text{s}$ . Therefore, with sealing temperature of 470°C, the sealing performance can meet the package requirement.

#### 4. Conclusions

The way for improving the life-cycle of vacuum glazing is a great challenge for vacuum glazing fabrication. Since the bonding strength of vacuum glazing plays an important role in the life-cycle of vacuum glazing, improving the bonding strength is a crucial matter for improving the durability performance of vacuum glazing. So, the research purpose of this paper is to improve the bonding strength of vacuum glazing and maximize the bonding strength for a particular bonding temperature. It is the first time that the anodic bonding method is used to help seal vacuum glazing. This method not only enhances the bonding strength of vacuum glazing, but also predigest the production process and reduce the fabrication costs. This work provides important theoretical basis and guidance for manufacturing and industrialization of the vacuum glazing and its application.

This study found that anodic bonding has a dramatic influence on sealing performance of the bond for vacuum glazing. When the sealing temperature and bonding time are set at certain values, with increasing the bonding voltage, the shear strength of sample increases.

With the sealing temperature of 450°C, the bonding time of 20min and the bonding voltage of 600 V, the shear strength is 2.585MPa, which is 2 times larger than that when no voltage was applied at the sample (1.082MPa). Elements such as Si, Ca, V, P and a small amount of Te elements were found at the interface between plane glass sheet and low-melting glass coating. This is due to the migration of ions in the glass activated by heat and voltage, forming a thin "intermediate composition" at the interface between the low temperature bonding and the surfaces of the glass sheets. With the temperature increasing, the migration actions accelerated, and the bond becomes stronger. With the temperature of 470°C, the bonding time of 20min and the bonding voltage of 600V, the shear

strength is 3.421MPa. The leakage rate is  $3.9 \times 10^{-9} \text{Pa} \cdot \text{m}^3/\text{s}$ , which meet the requirement of vacuum glazing.

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## Tables

**Table 1.** Components and various performance indexes of V<sub>2</sub>O<sub>5</sub>-P<sub>2</sub>O<sub>5</sub>-TeO<sub>2</sub> low-melting glass

Composition/wt%	V <sub>2</sub> O <sub>5</sub>	P <sub>2</sub> O <sub>5</sub>	TeO <sub>2</sub>	B <sub>2</sub> O <sub>3</sub>	ZnO	Na <sub>2</sub> O
	64.0	25.0	5.0	2.0	1.5	2.5
Properties	Sintering temperature		Glass transition temperature		Coefficient of thermal expansion	
	369.8 °C		339.3 °C		83.5 °C <sup>-1</sup>	

**Table 2.** The shear strength of samples at different bonding temperature

Sample no.	Temperature (°C)	Voltage (V)	Time (min)	Strength (MPa)
1	430	400	10	0.621
2	450	400	10	0.715
3	470	400	10	1.748
4	430	400	20	1.240
5	450	400	20	2.032
6	470	400	20	2.686

**Table 3.** The shear strength of samples at different bonding time

Sample no.	Temperature (°C)	Voltage (V)	Time (min)	Strength (MPa)
7	430	600	10	1.099
8	430	600	20	1.793
9	450	600	10	1.491
10	450	600	20	2.685
11	470	600	10	2.234
12	470	600	20	3.421

**Table 4.** The shear strength of samples at different bonding voltage

Sample no.	Temperature (°C)	Voltage (V)	Time (min)	Strength (MPa)
13	430	0	20	0.192
2	430	400	20	1.240
8	430	600	20	1.793
14	450	0	20	1.082
3	450	400	20	2.032
10	450	600	20	2.585
15	470	0	20	2.655
4	470	400	20	2.686
12	470	600	20	3.421

## Figure Captions

**Fig.1.** Schematic diagram of vacuum glazing

**Fig.2.** The sealing progress of vacuum glazing. (a) Printing glass paste. (b) Drying the sample and organic solvent is evaporated. (c) sealed the sample at the anodic bonding furnace.

**Fig.3.** The comparison of shear strength of the samples at different bonding temperatures

**Fig.4.** The picture of glass Ultra-depth three-dimension microscope picture at different bonding temperatures ( $\times 500$ )

**Fig.5.** The comparison of shear strength of the samples at different bonding time

**Fig.6.** The Ultra-depth three-dimension microscope picture at different bonding time ( $\times 500$ )

**Fig.7.** The comparison of shear strength of the samples at different bonding voltage

**Fig.8.** SEM micrograph of cross sectional of sealing samples sealed at 450°C, 20min, (a) 0V; (b)600V



(C) the EDS analysis of the low-melting coating(point ① and point ③) and plane sheet(point ② and point ④)

**Fig.9.** Microstructure of interface between low-melt glass and plane sheet. (a)SEM image, (b) element distribution curve of (a)

**Fig.10.**The schematic of the process of anodic bonding mechanism. (1 and 2 layer are the intermediate composition, **R** is the metal atom)

### **Table captions**

**Table 1.** Components and various performance indexes of  $V_2O_5$ - $P_2O_5$ - $TeO_2$  low-melting glass

**Table 2.** The shear strength of samples at different bonding temperature

**Table 3.** The shear strength of samples at different bonding time

**Table 4.** The shear strength of samples at different bonding voltage